


## Elastic gap-filling composition

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**Inventor:**  
**Applicant:** DEGUSSA  
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### Abstract of GB1083486

An elastic gap-filling composition is produced by polymerizing a monomeric methacrylate ester and at least one acrylate ester in the presence of at least one methacrylate ester/acrylate ester copolymer, and at least one paraffin wax soluble in the mixture, using an initiator effective at - 10 DEG to 50 DEG C. At least one cross-linking agent may also be included. Specified methacrylates are methyl, i-propyl, i-butyl, n-butyl and dodecyl methacrylate and the preferred acrylates have 3 to 18 carbon atoms in the ester group, e.g. linear and/or branched butyl, hexyl, octyl, 2-ethylhexyl, decyl, dodecyl or stearyl acrylates. The preformed copolymer may be present as a monomer/polymer syrup. Cross-linking agents specified are di (meth)acrylates of a dihydric alcohol such as ethylene, diethylene, triethylene, polyethylene, propylene, dipropylene, butylene or hexylene glycols, a divinyl compound, e.g. divinyl benzene and diallyl compounds, e.g. diallyl phthalate. Suitable catalysts are redox ones comprising a peroxide, preferably acyl, and a tertiary amine, preferably aromatic, with or without other accelerators, promoters and co-catalysts. Organic sulphur compounds, preferably mercaptans, may be used as modifiers. Suitable plasticizers, stabilizers and fillers are listed and pigments may also be included in the reaction mixture. In Example 1, 2-ethylhexyl methacrylate and methyl methacrylate are mixed with a n-butyl acrylate/methyl methacrylate copolymer, ethylene glycol dimethacrylate, lauryl mercaptan, paraffin wax, dimethyl aniline, a solution of benzoyl peroxide and dibutyl phthalate, crushed slate and silica and polymerized in a gap between two concrete sections. In Example 2, methyl methacrylate and n-butyl acrylate are mixed with a 2-ethylhexyl acrylate/methyl methacrylate copolymer, a chlorinated diphenyl, triphenyl phosphite, 2-(21-hydroxy-51-methylphenyl)-benzotriazole, dimethyl-p-toluidine, triethylene glycol diacrylate, paraffin wax, the aforesaid peroxide solution, BaSO<sub>4</sub> and SiO<sub>2</sub> and polymerized between two structural components consisting of foam concrete externally lined with P.V.C.-coated steel. 2-Ethylhexyl acrylate and methyl methacrylate are mixed in Example 3 with a methyl methacrylate/ethyl acrylate copolymer, a polyester of adipic acid and butylene glycol, dimethyl-p-toluidine, triethylene glycol dimethacrylate, paraffin wax, the catalyst mixture as in Example 1, crushed slate and SiO<sub>2</sub> and polymerized in a crack in a concrete tank. In Example 4, 2-ethylhexyl acrylate and methyl methacrylate are mixed with methyl methacrylate/2-ethylhexyl acrylate and n-butyl methacrylate/methyl methacrylate pre-polymers, triethylene glycol dimethacrylate, dimethyl p-toluidine, lauryl mercaptan, paraffin wax, the catalyst system of Example 1, and kaolin, and polymerized in joints between P.V.C.-lined sheet-steel tiles.

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# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### Elastic Gap-filling Composition

- We, DEUTSCHE GOLD-UND SILBER-SCHNEIDANSTALT VORMALS ROESSLER, of 9, Weissfrauenstrasse, Frankfurt (Main), Germany, a body corporate organised under the laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- 10 This invention relates to an elastic gap-filling compound.
- In buildings such as houses, factory, premises, facades, dams, locks, swimming pools, on roads, motorways and airport run-ways, in canals and sewage pipes and in fuel oil tanks, structural components which are movable relative to one another and move either by thermal expansion, soil subsidence or vibration, have to be joined together in such a way that, although the joints between them remain impermeable both to liquids and vapours, their limited mobility remains unaffected. This difficulty is overcome by using sealing compounds which, initially, are fluid and then become plastic or elastic, or permanently elastic materials in the form of profiles. Known sealing compounds include bitumen-based compositions, polysulphide rubber, polyurethanes, silicone rubber, and epoxide compositions. These materials are attended by various disadvantages such as brittleness at low temperatures, inadequate adhesion to various structural materials, low resistance both to acids and alkalis, deterioration of both the bond and the seal under the influence of water, poor ageing resistance or even a high price which rules out any possibility of the compounds being used for normal purposes.
- 40 Pre-formed seals such as rubber profiles of all kinds, profiles of polychloroprene, of chlorosulphonated polyethylene or of ethylene/propylene copolymers, which are vul-
- canised or crosslinked in known manner, profiles of soft PVC, composite materials made from fibres and plastics or rubber, are all attended by the common disadvantage that the shape has to be pre-determined with the result that an unexpectedly widened gap cannot be effectively filled. Although gap-filling compounds form only a small part of the total mass of a building of the aforementioned kind, it is extremely important that they should be both correct and reliable in function because a considerable proportion of all structural damage can be attributed to defective jointing or gap-filling.
- A gap-filling compound has to satisfy very stringent requirements. For example, it must be usable *in situ* and, initially, must be so highly fluid that it is able to penetrate into all the porous areas around the gap. At the same time, it must be thixotropically adjustable so that it does not run off into deeper lying cavities. In addition, it must become elastic after a period which, though short, is long enough for the compound to be worked and, at the same time, must not be affected by air or by the adjacent structural material (which may contain alkali). After it has become plastic, the compound must further be soft enough to enable the structural components on either side of it to make relatively quick movements without tearing the joint and, in addition has to bond firmly even to smooth surfaces.
- It has now been found that elastic gap-filling compounds based on polymerisable organic compounds which meet the requirements set out above, can be prepared by polymerising a mixture which consists essentially of a monomeric methacrylate ester, at least one acrylate ester, at least one copolymer of a methacrylate ester and an acrylate ester, and at least one paraffin wax soluble in the mixture and optionally at least one cross-linker with

an initiator or initiator system known *per se* which is effective at temperatures of from  $-10^{\circ}\text{C}$  to  $50^{\circ}\text{C}$ . The paraffin wax is preferably added in an amount of 0.01 to 5% by weight.

Apart from methyl methacrylate, the higher esters of methacrylic acid such as i-propyl, i-butyl, n-butyl and dodecyl esters, may also be used as the monomeric methacrylate esters and the methacrylate esters which form part of the copolymer.

The preferred monomeric acrylate esters and the acrylate esters which form part of the copolymer are those with an alkyl component containing from 3 to 18 carbon atoms. Basically, however, there is no upper limit to the length of this carbon chain. The alcohol radicals may be linear and/or branched. Examples of such compounds include butyl acrylate, hexyl acrylate, octyl acrylate, 2-ethylhexyl acrylate, decyl acrylate, dodecyl acrylate and stearyl acrylate. The acrylate ester may be the same in both cases.

For the purposes of this invention, it is preferred to use copolymers in the form of a pre-polymer prepared by partial polymerisation, as known *per se*. In this case, the copolymer is present in solution in the monomeric mixture. If desired, the consistency of these syrupy pre-polymers may be subsequently varied by adding more monomer.

Compounds containing two or more polymerisable double bonds in the molecule, may be used as the crosslinkers in known manner. One such compound may for example be a dimethacrylate or diacrylate of a dihydric alcohol such as ethylene glycol, diethylene glycol, triethylene glycol, polyethylene glycol, propylene glycol, dipropylene glycol, butylene glycol, hexylene glycol, a divinyl compound such as divinyl benzene or a diallyl compound such as diallyl phthalate.

Suitable polymerisation catalysts are those known compounds which initiate polymerisation at temperatures of from  $-10$  to  $50^{\circ}\text{C}$  in periods of a few hours. So-called redox catalysts comprising an organic peroxide, at least one tertiary amine and, where required, other accelerators and/or promoters and/or co-catalysts known *per se*, are preferably used. Of the tertiary amines, those whose nitrogen atoms are linked directly with at least one aromatic radical, are preferred. Of the peroxides, the acyl peroxides are preferred. Further constituents of these catalyst systems include organic sulphur compounds, preferably mercaptans.

Fillers and/or dyes and/or plasticisers and/or stabilisers may also be added to the compounds according to the invention.

The stabilisers which are intended to increase the resistance of the compounds to weathering, include, for example, the esters of phosphorous acid and UV-stabilisers, for

example benzophenone or benzotriazole derivatives.

The plasticisers which may be present in the mixtures according to the invention, include liquid esters of polyvalent acids such as phthalates, adipates, sebacates and, in particular, chlorinated di-, tri- and polyphenols, esters of phosphoric acid and chloroparaffins. It is also possible to use polymeric plasticisers such as, for example, esters of polybasic acids with polyhydric alcohols.

The elastic gap-filling compounds according to the invention may be mixed with fillers such as quartz power, crushed slate, asbestos powder and asbestos fibre, barium sulphate, readily soluble powdered plastics compositions, short glass or synthetic fibres. It is of greater advantage, however, to add pyrogenically prepared silicas which produce the required degree of thixotropy.

Any dyes which are fast to light and which may also be used to dye acrylic plastics may be used as the pigments providing they do not interfere with polymerisation.

The constituents used in the mixture as a whole are preferably employed in the following quantitative proportions which are by weight: methacrylate: 5 to 80%, preferably 30 to 60%; acrylate: 10 to 90%, preferably 20 to 70%; polymer content of the mixture as a whole: 10 to 80%, preferably 15 to 60; crosslinker: 0.01 to 20%, preferably 0.1 to 5%; paraffin wax: 0.01 to 5%, preferably 0.2 to 1.5%; fillers and pigments: 1 to 50%, preferably 1 to 25%.

In most cases, the gap-filling compound according to the invention may be used by mixing the required quantity with the initiator and applying the resulting mixture to the dried walls defining the gap with the tools normally used for such purposes. On the other hand, the compound could be sprayed into the gap. Precautions should be taken in every case to ensure that the area covered by the composition on the walls defining the gap, is sufficiently wide. The gaps themselves may run either vertically or horizontally. Vertical gaps can be filled by spraying providing the composition has been thixotropically adjusted to the required level.

The invention is further illustrated by the following Examples in which all parts and percentages are by weight:

#### EXAMPLE 1

60 parts of a pre-polymer prepared from 60 parts of methyl methacrylate and 40 parts of n-butyl acrylate (viscosity 2000 cP at  $20^{\circ}\text{C}$ ) were mixed with 20 parts of 2-ethylhexyl acrylate, 10 parts of methyl methacrylate, 6.5 parts of ethylene glycol dimethacrylate, 0.5 parts of lauryl mercaptan, 0.5 parts of paraffin wax, (m.p.  $52-54^{\circ}\text{C}$ ), 0.5 parts of dimethyl aniline and 2 parts of a 50% solution of benzoyl peroxide in dibutyl phthalate

(this component was added shortly before use).

- 15 parts of crushed slate and 2% of Aerosil (the word "Aerosil" being a Registered Trade Mark) were then mixed with the composition. After it has been introduced into a dried gap between two concrete motorway sections, the composition solidifies in one hour at 20° C. to form a tough material which bonds firmly to the concrete.

#### EXAMPLE 2

- 70 parts of a pre-polymer of 50 parts of 2-ethylhexyl acrylate and 50 parts of methyl methacrylate (viscosity 1800 cP) were mixed with 10 parts of methyl methacrylate, 10 parts of n-butyl acrylate, 5 parts of a chlorinated diphenyl (chlorine content about 60%), 1 part of triphenyl phosphite, 0.1 parts of 2(2' - hydroxy - 5' - methylphenyl) - benzotriazole, 0.3 parts of dimethyl-p-toluidine, 2 parts of triethylene glycol diacrylate and 0.5 parts of paraffin wax (m.p. 48—50°C).

- Shortly before the composition was used, 1.1 parts of benzoyl peroxide (50% in dibutyl phthalate) were added.

- The composition was mixed with 20 parts of barium sulphate and 2.5 parts of Aerosil. The composition was then used at a temperature of 20°C. to seal a gap between two structural components of a house. The components consist of foam concrete externally lined with PVC-coated steel. Following polymerisation, the gap-filling compound bonds firmly to the layer of PVC, the chamfered steel sheet and to the foam concrete.

#### EXAMPLE 3

- 50 parts of a prepolymer prepared from 80 parts of methyl methacrylate and 20 parts of ethyl acrylate (viscosity 3000 cP) were mixed with 25 parts of 2-ethylhexyl acrylate, 10 parts of methyl methacrylate, 10 parts of a polyadipate of adipic acid and butylene glycol (ABG), 3 parts of triethylene glycol dimethacrylate, 0.5 parts of paraffin wax (m.p. 54°C) and 0.5 parts of dimethyl-p-toluidine.

- Shortly before the composition was used, 1 part of benzoyl peroxide (in the form of a 50% solution in dibutyl phthalate) was added to it. Following the addition of 3 parts of Aerosil and 10 parts of crushed slate, the crack in a concrete fuel oil storage tank was filled and sealed with the composition. The composition polymerised over a period of some 2 hours at 20°C and bonded firmly to the concrete.

#### EXAMPLE 4

- 60 parts of a pre-polymer prepared from

50 parts of methyl methacrylate and 50 parts of 2-ethylhexyl acrylate (viscosity 1700 cP) were mixed with 10 parts of a prepolymer prepared from 70 parts of n-butyl methacrylate and 30 parts of methyl methacrylate (viscosity 2000 cP), and with 10 parts of 2-ethylhexyl acrylate, 10 parts of methyl methacrylate, 5 parts of triethylene glycol dimethacrylate, 1 part of dimethyl - p - toluidine, 0.5 parts of lauryl mercaptan, 0.5 parts of paraffin wax, m.p. 52°C, 3 parts of a 50% solution of benzoyl peroxide in dibutyl phthalate (added shortly before the composition is used). 20 parts of kaolin were added to the composition and it was used to fill, and thus permanently seal, the sickle-shaped, riveted joints in a roof made of PVC-lined sheet steel tiles. Polymerisation is complete after 1 hour at 20°C.

#### WHAT WE CLAIM IS:—

1. A process for the production of an elastic gap-filling composition based on polymerisable organic compounds, wherein a mixture which consists essentially of a monomeric methacrylate ester, at least one acrylate ester, at least one methacrylate ester/acrylate ester copolymer and at least one paraffin wax soluble in the mixture is polymerised with an initiator or initiator system known *per se* which is effective at temperatures of from -10°C to 50°C.
2. A process according to Claim 1 wherein from 0.1 to 5% by weight of paraffin wax is added.
3. A process according to Claim 1 or 2 wherein the mixture also includes a cross linking agent.
4. A process as claimed in Claim 1, wherein an acrylate whose alcohol component contains 3 to 18 carbon atoms is used.
5. A process as claimed in Claim 1 or 2, wherein the copolymer is used in the form of a pre-polymer prepared by partial polymerisation.
6. A process as claimed in any of Claims 1 to 5, wherein an organic peroxide, in combination with a tertiary amine and, where required, other accelerators and/or promoters and/or co-catalysts, in particular organic sulphur compounds, preferably mercaptans, is used as the initiator as known *per se*.
7. A process as claimed in Claims 1 to 6, wherein the mixture additionally contains fillers and/or dyes and/or plasticisers and/or stabilisers.
8. A process for the production of an elastic gap-filling composition substantially as described with reference to any of the Examples.

9. An elastic gap-filling composition produced by the process claimed in any of the preceding claims.

ELKINGTON & FIFE.  
Chartered Patent Agents,  
High Holborn House,  
52—54 High Holborn,  
London, W.C.1.  
Agents for the Applicants.

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